STIC Search Report
Biotech-Chem Library

STIC Database Tracking Number: 188062

TO: Rei-Tsang Shiao **Location: REM-5A10/5C18** **Art Unit: 1626** **Friday, May 12, 2006** **Case Serial Number: 10/811727**	**From: Paul Schulwitz** **Location: Biotech-Chem Library** **REM-1A65** **Phone: 571-272-2527** **Paul.schulwitz@uspto.gov**

Search Notes

Examiner Shiao,

Please review the attached search results.
If you have any questions or if you would like to refine the search query, please feel free to contact me at any time.

Thank you for using STIC search services!

Paul Schulwitz
Technical Information Specialist
REM-1A65
571-272-2527

STIC SEARCH RESULTS FEEDBACK FORM

Biotech-Chem Library

Questions about the scope or the results of the search? Contact ***the searcher or contact*:**

Mary Hale, Information Branch Supervisor
Remsen Bldg. 01 D86
571-272-2507

Voluntary Results Feedback Form

- *I am an examiner in Workgroup:* ☐ *Example: 1610*
- *Relevant prior art **found**, search results used as follows:*
  - ☐ 102 rejection
  - ☐ 103 rejection
  - ☐ Cited as being of interest.
  - ☐ Helped examiner better understand the invention.
  - ☐ Helped examiner better understand the state of the art in their technology.

Types of relevant prior art found:

- ☐ Foreign Patent(s)
- ☐ Non-Patent Literature
  (journal articles, conference proceedings, new product announcements etc.)

- *Relevant prior art **not found:***
  - ☐ Results verified the lack of relevant prior art (helped determine patentability).
  - ☐ Results were not useful in determining patentability or understanding the invention.

Comments:

Drop off or send completed forms to STIC-Biotech-Chem Library Remsen Bldg.

FOR OFFICIAL USE ONLY 188062 ACCESS DB # ____________

PLEASE PRINT CLEARLY

Scientific and Technical Information Center

SEARCH REQUEST FORM

Requester's Full Name: Robert (Reitsy) Shiao Examiner # : 79521 Date: 5/1/06

Art Unit: 1626 Phone Number: 2-0707 Serial Number: 10/811,727

Location (Bldg/Room#): REM (Mailbox #): 5A10 / 5C18 Results Format Preferred (circle): PAPER DISK

****

To ensure an efficient and quality search, please attach a copy of the cover sheet, claims, and abstract or fill out the following:

Title of Invention: gamma crystalline form

Inventors (please provide full names): pfeiffer et al.

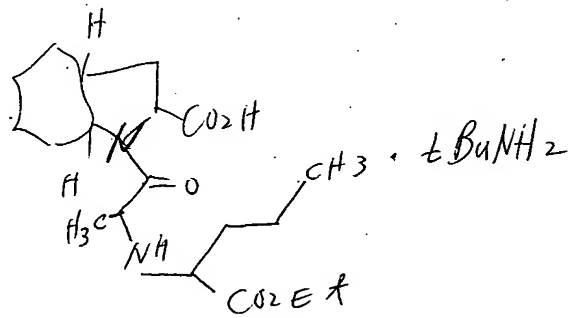
Earliest Priority Date: ____________

Search Topic:

Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc., if known.

For Sequence Searches Only ***Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.***

I. search cpd z : (see claim 12)



II search crystalline form of cpd z.

INV L15

L30

(UE)

7:0 30 $31.51

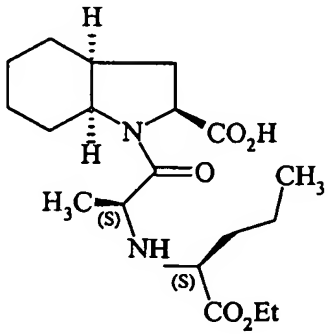
****

STAFF USE ONLY	Type of Search	Vendors and cost where applicable	
Searcher: ____	____ NA Sequence (#)	____ STN	____ Dialog
Searcher Phone #: ____	____ AA Sequence (#)	____ Questel/Orbit	____ Lexis/Nexis
Searcher Location: ____	____ Structure (#)	____ Westlaw	____ WWW/Internet
Date Searcher Picked Up: ____	____ Bibliographic	____ In-house sequence systems	
Date Completed: ____	____ Litigation	____ Commercial ____ Oligomer ____ Score/Length	
Searcher Prep & Review Time: ____	____ Fulltext	____ Interference ____ SPDI ____ Encode/Transl	
Online Time: ____	____ Other	____ Other (specify)	

LISTING OF CLAIMS

1-11. (canceled)

12. (previously presented) A γ crystalline form of the compound of formula (I) :



. $tBuNH_2$ (I),

exhibiting essentially the following powder X-ray diffraction data, measured using a diffractometer (copper anticathode) and expressed in terms of inter-planar distance d, Bragg's angle 2 theta, intensity and relative intensity (expressed as a percentage with respect to the most intense ray) :

Angle 2 theta (°)	Inter-planar distance d (Å)	Intensity	Relative intensity (%)
6.298	14.02	630	39.8
7.480	11.81	380	24
8.700	10.16	1584	100
9.276	9.53	318	20.1
10.564	8.37	526	33.2
11.801	7.49	54	3.4
12.699	6.96	86	5.4
13.661	6.48	178	11.2
14.095	6.28	163	10.3
14.332	6.17	290	18.3
14.961	5.92	161	10.2
15.793	5.61	128	8.1
16.212	5.46	179	11.3
16.945	5.23	80	5.1

17.291	5.12	92	5.8
17.825	4.97	420	26.5
18.100	4.90	159	10
18.715	4.74	89	5.6
19.017	4.66	118	7.4
19.362	4.58	134	8.5
19.837	4.47	133	8.4
20.609	4.31	95	6
21.232	4.18	257	16.2
21.499	4.13	229	14.5
21.840	4.07	127	8
22.129	4.01	191	12.1
22.639	3.92	137	8.6
23.000	3.86	88	5.6
23.798	3.74	147	9.3
24.170	3.68	70	4.4
25.066	3.55	167	10.5
25.394	3.50	165	10.4
26.034	3.42	84	5.3
26.586	3.35	75	4.7
27.541	3.24	74	4.7
28.330	3.15	85	5.4
29.589	3.02	96	6.1

13. (previously presented) A process for the preparation of the γ crystalline form of the compound of claim 12, wherein a solution of perindopril tert-butylamine salt in chloroform is heated at reflux, the solution is then cooled to 0°C and the solid obtained is collected by filtration.

14. (previously presented) A process for the preparation of the γ crystalline form of the compound of claim 12, wherein a solution of perindopril tert-butylamine salt in ethyl acetate is heated at reflux, the solution is rapidly cooled, the solid thereby obtained is then collected by filtration, it is suspended in chloroform, the suspension is stirred at ambient temperature for 5 to 10 days, and the solid is then collected by filtration.

15. (previously presented) The process of claim 13, wherein the compound of formula (I) obtained by the preparation process described in patent specification EP 0 308 341 is used.

16. (previously presented) The process of claim 13, wherein the concentration of the compound of formula (I) in the chloroform is 150 to 300 g/litre.

17. (previously presented) The process of claim 14, wherein the compound of formula (I) obtained by the preparation process described in patent specification EP 0 308 341 is used.

18. (previously presented) The process according to claim 14, wherein the concentration of the compound of formula (I) in the ethyl acetate is 70 to 90 g/litre.

19. (previously presented) A method of treating a living animal body afflicted with a condition requiring an inhibitor of angiotensin I converting enzyme, comprising the step of administering to the living animal body an amount of the compound of claim 12 which is effective for alleviation of the condition.

20. (previously presented) A pharmaceutical composition comprising, as active principle, an effective amount of the compound of claim 12, together with one or more pharmaceutically acceptable excipients or vehicles.

21. (previously presented) A method of treating a living animal body afflicted with a cardiovascular disease, comprising the step of administering to the living animal body an amount of the compound of claim 12 which is effective for alleviation of the condition.

22. (previously presented) The pharmaceutical composition of claim 20, which also comprises a diuretic.

23. (previously presented) The pharmaceutical composition of claim 22, wherein the diuretic is indapamide.

UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

Bib Data Sheet

CONFIRMATION NO. 1430

SERIAL NUMBER	FILING DATE	CLASS	GROUP ART UNIT	ATTORNEY DOCKET NO.
10/811,727	03/29/2004 RULE	548	1626	SERVIER 398 PCT

APPLICANTS

Bruno Pfeiffer, Saint Leu La Foret, FRANCE;

Yves-Michel Ginot, Orleans, FRANCE;
Gerard Coquerel, Boos, FRANCE;Stephane Beilles, Dijon, FRANCE;

** CONTINUING DATA **********
This application is a CON of 10/312,903 12/31/2002 ABN

** FOREIGN APPLICATIONS **********
FRANCE 00.08791 07/06/2000
FRANCE PCT/FR01/02169 07/06/2001

IF REQUIRED, FOREIGN FILING LICENSE GRANTED
** 06/07/2004

Foreign Priority claimed	35 USC 119 (a-d) conditions met	Verified and Acknowledged	STATE OR COUNTRY	SHEETS DRAWING	TOTAL CLAIMS	INDEPENDENT CLAIMS
☒ yes ☐ no	☒ yes ☐ no ☐ Met after Allowance	Examiner's Signature Initials	FRANCE	0	12	1

ADDRESS
The Firm of Hueschen and Sage
500 Columbia Plaza
350 East Michigan Avenue
Kalamazoo , MI
49007

TITLE
Gamma crystalline form of perindopril tert-butylamine salt

FILING FEE	FEES: Authority has been given in Paper	☐ All Fees ☐ 1.16 Fees (Filing) ☐ 1.17 Fees (Processing Ext. of

Application Search

L3 ANSWER 1 OF 1 HCAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 2001:816626 HCAPLUS
DOCUMENT NUMBER: 135:344373
ENTRY DATE: Entered STN: 09 Nov 2001
TITLE: Process for preparing the novel γ crystalline form of the diuretic perindopril tert-butylamine salt
INVENTOR(S): Pfeiffer, Bruno; Ginot, Yves-Michel; Coquerel, Gerard; Beilles, Stephane
PATENT ASSIGNEE(S): Adir et Compagnie, Fr.
SOURCE: PCT Int. Appl., 11 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: French
INT. PATENT CLASSIF.:
MAIN: C07D
CLASSIFICATION: 27-11 (Heterocyclic Compounds (One Hetero Atom))
Section cross-reference(s): 63, 75
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2001083439	A2	20011108	WO 2001-FR2169	20010706
WO 2001083439	A3	20020207		
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
FR 2811318	A1	20020111	FR 2000-8791	20000706
FR 2811318	B1	20020823		
CA 2415447	AA	20011108	CA 2001-2415447	20010706
AU 2001076420	A5	20011112	AU 2001-76420	20010706
EP 1296948	A2	20030402	EP 2001-954060	20010706
EP 1296948	B1	20030910		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
BR 2001012211	A	20030506	BR 2001-12211	20010706
AT 249435	E	20030915	AT 2001-954060	20010706
JP 2003531890	T2	20031028	JP 2001-580868	20010706
JP 3592296	B2	20041124		
PT 1296948	T	20031231	PT 2001-954060	20010706
ES 2206423	T3	20040516	ES 2001-1954060	20010706
NZ 523311	A	20040625	NZ 2001-523311	20010706
EE 200300003	A	20040816	EE 2003-3	20010706
US 2003158121	A1	20030821	US 2002-312903	20021231
ZA 2003000025	A	20040210	ZA 2003-25	20030102
NO 2003000051	A	20030106	NO 2003-51	20030106
BG 107534	A	20031231	BG 2003-107534	20030205
HR 2003000078	A1	20030430	HR 2003-78	20030206
HR 20030078	B1	20040630		
US 2004248817	A1	20041209	US 2004-811727	20040329 <--
JP 2005002120	A2	20050106	JP 2004-206157	20040713
PRIORITY APPLN. INFO.:			FR 2000-8791 A	20000706

JP 2001-580868	A3	20010706
WO 2001-FR2169	W	20010706
US 2002-312903	B1	20021231

PATENT CLASSIFICATION CODES:

PATENT NO.	CLASS	PATENT FAMILY CLASSIFICATION CODES
WO 2001083439	ICM	C07D
	IPCI	C07D [ICM,7]
	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
	ECLA	C07D209/42
FR 2811318	IPCI	C07D0209-42 [ICM,7]; A61K0031-405 [ICS,7]; A61P0009-00 [ICS,7]; A61P0007-10 [ICS,7]; A61K0031-405 [ICI,7]; A61K0031-4045 [ICI,7]
	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
	ECLA	C07D209/42
CA 2415447	IPCI	C07D0209-42 [ICM,7]; A61P0009-00 [ICS,7]; A61K0031-404 [ICS,7]
	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
AU 2001076420	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
EP 1296948	IPCI	C07D0209-42 [ICM,7]
	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
BR 2001012211	IPCI	C07D0209-42 [ICM,7]
	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
AT 249435	IPCI	C07D0209-42 [ICM,7]
JP 2003531890	IPCI	C07D0209-42 [ICM,7]; A61K0031-404 [ICS,7]; A61K0045-00 [ICS,7]; A61P0009-10 [ICS,7]; A61P0013-02 [ICS,7]; A61P0043-00 [ICS,7]; B01D0009-02 [ICS,7]; C07M0007-00 [ICS,7]
	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
PT 1296948	IPCI	C07D0209-42 [ICM,7]
ES 2206423	IPCI	C07D0209-42 [ICM,7]
NZ 523311	IPCI	C07D0209-42 [ICM,7]; A61K0031-404 [ICS,7]; A61P0009-00 [ICS,7]
	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
EE 200300003	IPCI	A61K0031-404 [ICM,7]; A61P0009-00 [ICS,7]; C07D0209-42 [ICS,7]
US 2003158121	IPCI	A61K0038-04 [ICM,7]; C07D0209-12 [ICS,7]
	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
	NCL	514/019.000
	ECLA	C07D209/42
ZA 2003000025	IPCI	C07D [ICM,7]; A61K [ICS,7]; A61P [ICS,7]
NO 2003000051	IPCI	C07D0207-16 [ICM,7]
BG 107534	IPCI	C07D0209-42 [ICM,7]
HR 2003000078	IPCI	C07D0209-42 [ICM,7]; A61K0031-404 [ICS,7]; A61P0009-00 [ICS,7]
US 2004248817	IPCI	A61K0038-04 [ICM,7]; C07K0005-04 [ICS,7]; C07D0209-42 [ICS,7]
	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
	NCL	514/019.000
	ECLA	C07D209/42
JP 2005002120	IPCI	C07K0005-062 [ICM,7]; A61K0031-404 [ICS,7]; A61K0038-00 [ICS,7]; A61K0045-00 [ICS,7]; A61P0007-10 [ICS,7]; A61P0009-00 [ICS,7]; A61P0009-04 [ICS,7]; A61P0009-12 [ICS,7]; A61P0043-00 [ICS,7]; C07D0209-08 [ICS,7]
	IPCR	C07D0209-00 [I,C]; C07D0209-42 [I,A]
	FTERM	4C084/AA02; 4C084/AA03; 4C084/AA07; 4C084/AA19; 4C084/BA01; 4C084/BA14; 4C084/BA23; 4C084/CA59; 4C084/MA02; 4C084/NA14; 4C084/ZA361; 4C084/ZA362; 4C084/ZA421; 4C084/ZA422; 4C084/ZA832; 4C084/ZC202;

4C086/AA01; 4C086/AA02; 4C086/BC13; 4C086/MA02;
4C086/MA04; 4C086/NA05; 4C086/ZA36; 4C086/ZA42;
4C086/ZA83; 4C204/CB03; 4C204/DB03; 4C204/EB02;
4C204/FB35; 4C204/GB01; 4H045/AA10; 4H045/AA30;
4H045/BA11; 4H045/DA57; 4H045/EA23

ABSTRACT:
The γ crystalline form of the diuretic perindopril tert-butylamine salt (I) is prepared by refluxing a chloroform-I solution, cooling the solution to 0°, and filtering the I γ crystal modification which is characterized by its X-ray diffraction pattern; a I-containing formulation is presented.

SUPPL. TERM: diuretic perindopril tertiary butylamine salt crystal modification

INDEX TERM: Polymorphism (crystal)
(process for preparing the novel γ crystalline form of the diuretic perindopril tert-butylamine salt)

INDEX TERM: Cardiovascular agents
(process for preparing the novel γ crystalline form of the diuretic perindopril tert-butylamine salt for preparation of)

INDEX TERM: Cooling
Crystallization
Filtration
(process for preparing the novel γ crystalline form of the diuretic perindopril tert-butylamine salt using)

INDEX TERM: Separation
(reflux; process for preparing the novel γ crystalline form of the diuretic perindopril tert-butylamine salt using)

INDEX TERM: **9015-82-1**
ROLE: BSU (Biological study, unclassified); BUU (Biological use, unclassified); BIOL (Biological study); USES (Uses)
(inhibitors; process for preparing the novel γ crystalline form of the diuretic perindopril tert-butylamine salt for preparation of)

INDEX TERM: **107133-36-8**
ROLE: PEP (Physical, engineering or chemical process); PRP (Properties); PROC (Process)
(process for preparing the novel γ crystalline form of the diuretic perindopril tert-butylamine salt)

INDEX TERM: **484-42-4**
ROLE: BPR (Biological process); BSU (Biological study, unclassified); BIOL (Biological study); PROC (Process)
(process for preparing the novel γ crystalline form of the diuretic perindopril tert-butylamine salt as an intermediate in the preparation of inhibitors of)

INDEX TERM: **26807-65-8P**, Indapamide
ROLE: PNU (Preparation, unclassified); PREP (Preparation)
(process for preparing the novel γ crystalline form of the diuretic perindopril tert-butylamine salt for the preparation of)

INDEX TERM: **67-66-3**, Chloroform, uses **141-78-6**, Ethyl acetate, uses
ROLE: NUU (Other use, unclassified); USES (Uses)
(solvent; process for preparing the novel γ crystalline form of the diuretic perindopril tert-butylamine salt)

IT **9015-82-1**
RL: BSU (Biological study, unclassified); BUU (Biological use, unclassified); BIOL (Biological study); USES (Uses)
(inhibitors; process for preparing the novel γ crystalline form of the diuretic perindopril tert-butylamine salt for preparation of)

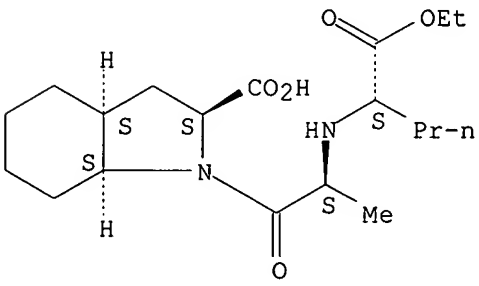
RN 9015-82-1 HCAPLUS
CN Carboxypeptidase, dipeptidyl, A (9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***
IT **107133-36-8**
RL: PEP (Physical, engineering or chemical process); PRP (Properties); PROC (Process)
(process for preparing the novel γ crystalline form of the diuretic perindopril tert-butylamine salt)
RN 107133-36-8 HCAPLUS
CN 1H-Indole-2-carboxylic acid, 1-[(2S)-2-[[(1S)-1-(ethoxycarbonyl)butyl]amino]-1-oxopropyl]octahydro-, (2S,3aS,7aS)-, compd. with 2-methyl-2-propanamine (1:1) (9CI) (CA INDEX NAME)

CM 1

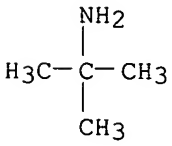
CRN 82834-16-0
CMF C19 H32 N2 O5

Absolute stereochemistry. Rotation (-).



CM 2

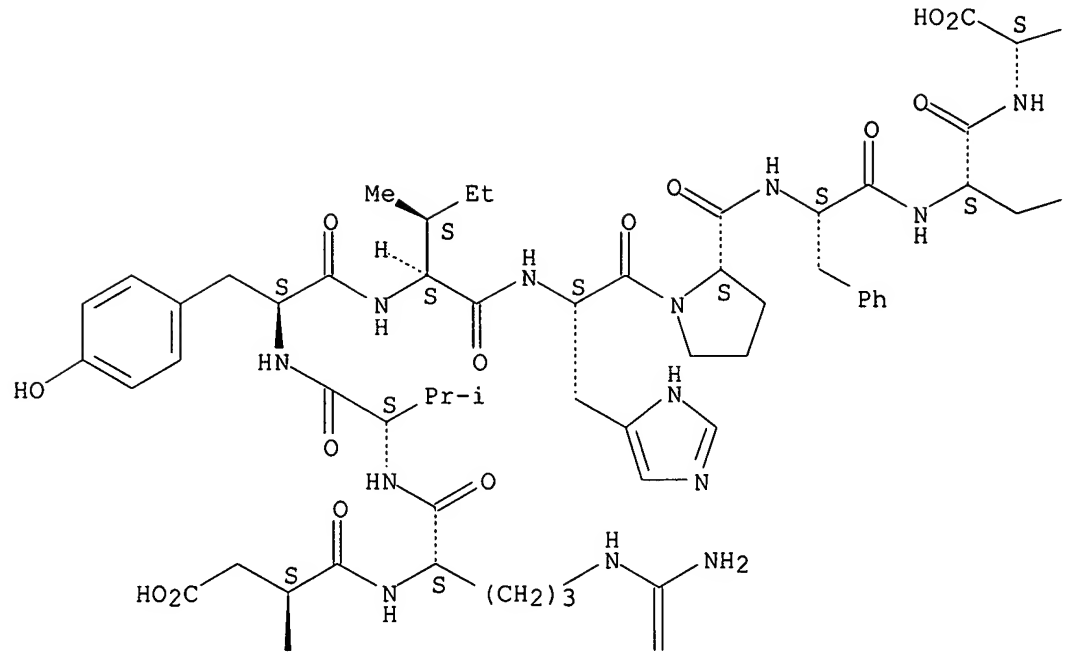
CRN 75-64-9
CMF C4 H11 N



IT **484-42-4**
RL: BPR (Biological process); BSU (Biological study, unclassified); BIOL (Biological study); PROC (Process)
(process for preparing the novel γ crystalline form of the diuretic perindopril tert-butylamine salt as an intermediate in the preparation of inhibitors of)
RN 484-42-4 HCAPLUS
CN Angiotensin I, 5-L-isoleucine- (8CI, 9CI) (CA INDEX NAME)

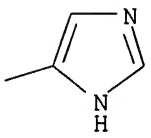
Absolute stereochemistry.

PAGE 1-A



PAGE 1-B

Bu-i

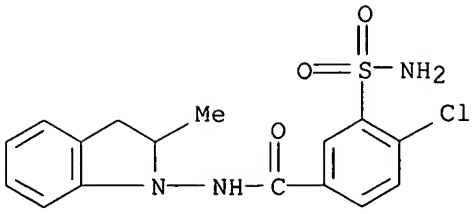


PAGE 2-A

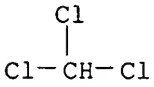
IT **26807-65-8P**, Indapamide
RL: PNU (Preparation, unclassified); PREP (Preparation)
(process for preparing the novel γ crystalline form of the diuretic perindopril tert-butylamine salt for the preparation of)

RN 26807-65-8 HCAPLUS

CN Benzamide, 3-(aminosulfonyl)-4-chloro-N-(2,3-dihydro-2-methyl-1H-indol-1-yl)- (9CI) (CA INDEX NAME)



IT **67-66-3**, Chloroform, uses **141-78-6**, Ethyl acetate, uses
RL: NUU (Other use, unclassified); USES (Uses)
(solvent; process for preparing the novel γ crystalline form of the diuretic perindopril tert-butylamine salt)
RN 67-66-3 HCAPLUS
CN Methane, trichloro- (9CI) (CA INDEX NAME)



RN 141-78-6 HCAPLUS
CN Acetic acid ethyl ester (8CI, 9CI) (CA INDEX NAME)